Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dipotassium hexafluoridozirconate(IV) hydrogen fluoride, $K_2ZrF_6{\cdot}HF$

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Received 11 July 2007; accepted 24 July 2007

Key indicators: single-crystal X-ray study; T = 203 K; mean σ(Zr–F) = 0.001 Å; R factor = 0.030; wR factor = 0.071; data-to-parameter ratio = 55.5.

The title compound is built from edge-sharing centrosymmetric dimeric $[Zr_2F_{12}]^{4-}$ complexes, K^+ cations and HF molecules. The Zr^{IV} coordination polyhedron is a distorted monocapped octahedron. The K^+ ions are coordinated by F atoms and HF molecules with potassium coordination numbers of 7 and 8. In addition, the structure is stabilized by strong F—H···F hydrogen bonds.

Related literature

For related literature, see: Blatov (2004); Serezhkin *et al.* (1997); Tkachev *et al.* (1993).

Experimental

Crystal data

$K_2ZrF_6{\cdot}HF$
M_r = 303.43
Orthorhombic, *Pbca*
a = 13.3641 (5) Å
b = 11.4457 (4) Å
c = 8.5211 (3) Å
V = 1303.40 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.03 mm^{-1}
T = 203 (2) K
0.44 × 0.37 × 0.27 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: Gaussian (*SADABS*; Bruker, 2003)
T_{min} = 0.443, T_{max} = 0.535
23595 measured reflections
5275 independent reflections
4834 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.071
S = 1.21
5275 reflections
95 parameters
Only H-atom coordinates refined
$\Delta\rho_{max}$ = 1.58 e Å^{-3}
$\Delta\rho_{min}$ = −1.30 e Å^{-3}

Table 1
Selected bond lengths (Å).

Zr—F5	1.9799 (7)	K1—F2^{v}	2.8115 (6)
Zr—F4	2.0043 (7)	K1—F7^{vi}	2.8284 (8)
Zr—F2	2.0241 (5)	K1—F3^{ii}	2.8805 (7)
Zr—F1	2.0290 (6)	K1—F7^{vii}	2.9848 (9)
Zr—F6	2.0790 (6)	K2—F2^{viii}	2.6365 (6)
Zr—F3	2.1696 (6)	K2—F1^{ix}	2.6506 (7)
Zr—F3^{i}	2.1833 (6)	K2—F5^{x}	2.6517 (7)
K1—F2^{ii}	2.6214 (6)	K2—F4^{viii}	2.6735 (7)
K1—F4^{iii}	2.6411 (7)	K2—F6^{v}	2.7822 (7)
K1—F5^{iv}	2.7079 (8)	K2—F1	2.8541 (6)
K1—F1^{v}	2.7232 (6)	K2—F7	2.8774 (9)

Symmetry codes: (i) $-x, -y, -z$; (ii) $x, y+1, z+1$; (iii) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$; (iv) $-x+\frac{1}{2}, y+\frac{1}{2}, z+1$; (v) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (vi) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$; (vii) $-x, -y+1, -z+1$; (viii) $-x+\frac{1}{2}, -y, z+\frac{1}{2}$; (ix) $-x, -y, -z+1$; (x) $x, y, z+1$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
F7—H1···F6	0.87 (2)	1.52 (2)	2.3865 (10)	172 (2)

Data collection: *SMART* (Bruker, 1998*a*); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1998*b*); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank the Russian Foundation for Basic Research (project No. 05-03-33298) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: MG2031).

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 doi:10.1107/S160053680703615X

supplementary materials

Acta Cryst. (2007). E**63**, i171 [**doi:10.1107/S160053680703615X**]

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Comment

The title compound, (I), is isostructural to $K_2Rb_2(ZrF_6)_2{\cdot}2H_2O$, (II), if hydrogen atoms are neglected (Tkachev *et al.*, 1993). The asymmetric unit contains one crystallographically independent Zr atom, six fluorine atoms, two potassium cations and one hydrogen fluoride molecule. The Zr atoms are coordinated by seven F atoms in a distorted monocapped octahedral geometry. Two Zr-centred polyhedra are linked by double F bridges to form a centrosymmetric dimeric $[Zr_2F_{12}]^{4-}$ complex (Fig. 1) with a $Zr\cdots Zr^{i}$ distance of 3.6810 (2) Å. Parallel to the *ac* plane, layers of K1 atoms and HF molecules alternate with layers of K2 atoms and $[Zr_2F_{12}]^{4-}$ complexes (Fig. 2).

The coordination numbers (CN) of potassium atoms were calculated by the method of intersecting spheres (Serezhkin *et al.*, 1997) with use of the program package *TOPOS* (Blatov, 2004). For K1, the CN is 8 (K1–F, 2.6214 (6)–2.9848 (9) Å) and for K2, the CN is 7 (K2–F, 2.6365 (6)–2.8774 (9) Å).

The HF molecule is coordinated to both potassium atoms and is involved as a donor in the strong hydrogen bond (2.3865 (10) Å) with the F6 atom (Fig. 2). Unlike the structure of (II), where the water molecule forms two hydrogen bonds with an F6 atom (2.88 Å) and a bridging F3 atom (2.74 Å) (Tkachev *et al.*, 1993), in (I) there is only one hydrogen bond, resulting in a lengthened Zr–F6 bond (2.0790 (6) Å) and nearly equal Zr–F3 and $Zr–F3^{i}$ bridging bonds (2.1696 (6) and 2.1833 (6) Å). In (II), the corresponding bonds are 2.046, 2.188, and 2.199 Å, respectively (Tkachev *et al.*, 1993).

Experimental

ZrO_2 (6.2 g, 0.05 mol) was reacted with KHF_2 (7.9 g, 0.1 mol) in a solution of hydrofluoric acid (40%, 30 ml). Upon slow evaporation at room temperature, crystals of K_2ZrF_6 precipitated first, and after they were separated, crystals of (I) precipitated next.

Refinement

The H1 atom was found in a difference Fourier map and refined with $U_{iso}(H1) = 1.5U_{eq}(F7)$. The maximum peak and deepest hole are located 0.51 Å and 0.97 Å, respectively, from Zr and K2.

Figures

- Fig. 1. A view of the centrosymmetric dimeric $[Zr_2F_{12}]^{4-}$ complex, with displacement ellipsoids drawn at the 50% probability level.
- Fig. 2. The structure of (I), viewed along the *c* axis.

Dipotassium hexafluoridozirconate(IV) hydrogen fluoride

Crystal data

$K_2ZrF_6 \cdot HF$	$F_{000} = 1136$
$M_r = 303.43$	$D_x = 3.093$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2ab	Cell parameters from 1939 reflections
$a = 13.3641$ (5) Å	$\theta = 3.6$–$45.0°$
$b = 11.4457$ (4) Å	$\mu = 3.03$ mm^{-1}
$c = 8.5211$ (3) Å	$T = 203$ (2) K
$V = 1303.40$ (8) Å^3	Prism, colourless
$Z = 8$	0.44 × 0.37 × 0.27 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	5275 independent reflections
Radiation source: fine-focus sealed tube	4834 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
Detector resolution: 8.33 pixels mm^{-1}	$\theta_{max} = 45.0°$
$T = 203(2)$ K	$\theta_{min} = 3.1°$
ω scans	$h = -26 \rightarrow 23$
Absorption correction: gaussian (SADABS in XPREP; Bruker, 2003)	$k = -20 \rightarrow 22$
$T_{min} = 0.443$, $T_{max} = 0.535$	$l = -16 \rightarrow 16$
23595 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	Only H-atom coordinates refined
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.0278P)^2 + 0.8258P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.071$	$(\Delta/\sigma)_{max} = 0.003$
$S = 1.21$	$\Delta\rho_{max} = 1.58$ e Å^{-3}
5275 reflections	$\Delta\rho_{min} = -1.30$ e Å^{-3}
95 parameters	Extinction correction: SHELXTL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0049 (2)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\mathrm{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zr	0.118901 (5)	0.040452 (6)	0.094477 (8)	0.00846 (1)
K1	0.170769 (16)	0.740441 (15)	0.84850 (2)	0.01749 (3)
K2	0.106835 (15)	0.06846 (2)	0.61088 (3)	0.01853 (3)
F1	0.08118 (5)	−0.02918 (5)	0.30531 (7)	0.01728 (10)
F2	0.20020 (4)	−0.10800 (5)	0.07447 (7)	0.01528 (9)
F3	0.04308 (4)	−0.06348 (6)	−0.08217 (8)	0.02043 (11)
F4	0.25374 (5)	0.09337 (6)	0.16899 (10)	0.02846 (15)
F5	0.15406 (7)	0.12846 (7)	−0.09782 (8)	0.02574 (14)
F6	0.08494 (5)	0.19919 (5)	0.20043 (8)	0.02147 (12)
F7	−0.03330 (6)	0.18673 (8)	0.40966 (9)	0.02981 (17)
H1	0.0108 (18)	0.198 (2)	0.335 (3)	0.045*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zr	0.00841 (2)	0.00835 (2)	0.00861 (3)	−0.00061 (2)	−0.00108 (2)	0.00001 (2)
K1	0.02166 (7)	0.01343 (5)	0.01738 (7)	−0.00241 (5)	0.00038 (5)	−0.00296 (5)
K2	0.01207 (6)	0.02388 (7)	0.01963 (7)	0.00100 (5)	−0.00039 (5)	−0.00405 (6)
F1	0.0187 (2)	0.0189 (2)	0.0143 (2)	0.00307 (17)	0.00298 (17)	0.00444 (16)
F2	0.01427 (19)	0.01355 (17)	0.0180 (2)	0.00254 (15)	−0.00253 (16)	−0.00328 (15)
F3	0.01218 (19)	0.0261 (2)	0.0230 (2)	0.00429 (18)	−0.00479 (17)	−0.01411 (19)
F4	0.0159 (2)	0.0211 (2)	0.0483 (4)	−0.00098 (19)	−0.0129 (3)	−0.0108 (3)
F5	0.0333 (3)	0.0260 (3)	0.0179 (3)	−0.0053 (3)	0.0052 (2)	0.0075 (2)
F6	0.0268 (3)	0.01294 (18)	0.0247 (3)	−0.00303 (18)	0.0042 (2)	−0.00626 (18)
F7	0.0254 (3)	0.0383 (4)	0.0258 (3)	0.0039 (3)	0.0074 (2)	−0.0036 (3)

Geometric parameters (Å, °)

Zr—F5	1.9799 (7)	K2—F4^{xiv}	2.6735 (7)
Zr—F4	2.0043 (7)	K2—F6^{x}	2.7822 (7)
Zr—F2	2.0241 (5)	K2—F1	2.8541 (6)
Zr—F1	2.0290 (6)	K2—F7	2.8774 (9)

Zr—F6	2.0790 (6)	K2—F7^{v}	3.0867 (10)
Zr—F3	2.1696 (6)	K2—F3^{xv}	3.1381 (7)
Zr—F3^{i}	2.1833 (6)	K2—K2^{v}	3.7657 (4)
Zr—K2^{ii}	3.8740 (2)	K2—Zr^{xiv}	3.8740 (2)
Zr—K1^{iii}	3.9373 (2)	K2—K1^{xvi}	4.0997 (3)
Zr—K1^{iv}	4.0823 (2)	F1—K2^{v}	2.6506 (7)
Zr—K2^{v}	4.1180 (2)	F1—K1^{iii}	2.7232 (6)
Zr—K2^{vi}	4.1364 (3)	F2—K1^{iv}	2.6214 (6)
K1—F2^{vii}	2.6214 (6)	F2—K2^{ii}	2.6365 (6)
K1—F4^{viii}	2.6411 (7)	F2—K1^{iii}	2.8115 (6)
K1—F5^{ix}	2.7079 (8)	F3—Zr^{i}	2.1833 (6)
K1—F1^{x}	2.7232 (6)	F3—K1^{iv}	2.8805 (7)
K1—F2^{x}	2.8115 (6)	F3—K2^{vi}	3.1381 (7)
K1—F7^{xi}	2.8284 (8)	F4—K1^{xvii}	2.6411 (7)
K1—F3^{vii}	2.8805 (7)	F4—K2^{ii}	2.6735 (7)
K1—F7^{xii}	2.9848 (9)	F4—K1^{xviii}	3.3629 (9)
K1—F4^{ix}	3.3629 (9)	F5—K2^{vi}	2.6517 (7)
K1—Zr^{x}	3.9373 (2)	F5—K1^{xviii}	2.7079 (8)
K1—Zr^{vii}	4.0823 (2)	F6—K2^{iii}	2.7822 (6)
K1—K2^{xiii}	4.0997 (3)	F7—K1^{xix}	2.8284 (8)
K2—F2^{xiv}	2.6365 (6)	F7—K1^{xii}	2.9848 (9)
K2—F1^{v}	2.6506 (7)	F7—K2^{v}	3.0867 (10)
K2—F5^{xv}	2.6517 (7)		
F5—Zr—F4	83.98 (4)	F7^{xi}—K1—Zr^{vii}	72.45 (2)
F5—Zr—F2	103.30 (3)	F3^{vii}—K1—Zr^{vii}	30.543 (12)
F4—Zr—F2	78.33 (3)	F7^{xii}—K1—Zr^{vii}	92.234 (18)
F5—Zr—F1	172.54 (3)	F4^{ix}—K1—Zr^{vii}	93.154 (13)
F4—Zr—F1	93.53 (3)	Zr^{x}—K1—Zr^{vii}	159.923 (6)
F2—Zr—F1	83.00 (2)	F2^{vii}—K1—K2^{xiii}	124.870 (15)
F5—Zr—F6	88.09 (3)	F4^{viii}—K1—K2^{xiii}	77.459 (17)
F4—Zr—F6	78.15 (3)	F5^{ix}—K1—K2^{xiii}	39.595 (15)
F2—Zr—F6	152.55 (3)	F1^{x}—K1—K2^{xiii}	79.959 (14)
F1—Zr—F6	84.52 (3)	F2^{x}—K1—K2^{xiii}	39.604 (12)
F5—Zr—F3	79.37 (3)	F7^{xi}—K1—K2^{xiii}	136.58 (2)
F4—Zr—F3	143.64 (3)	F3^{vii}—K1—K2^{xiii}	154.800 (16)
F2—Zr—F3	74.45 (2)	F7^{xii}—K1—K2^{xiii}	102.494 (16)
F1—Zr—F3	106.43 (3)	F4^{ix}—K1—K2^{xiii}	86.747 (13)
F6—Zr—F3	132.73 (3)	Zr^{x}—K1—K2^{xiii}	57.592 (4)
F5—Zr—F3^{i}	97.71 (3)	Zr^{vii}—K1—K2^{xiii}	141.310 (6)
F4—Zr—F3^{i}	150.48 (3)	F2^{xiv}—K2—F1^{v}	171.13 (2)

F2—Zr—F3^{i}	129.02 (2)	F2^{xiv}—K2—F5^{xv}	80.38 (2)
F1—Zr—F3^{i}	81.00 (3)	F1^{v}—K2—F5^{xv}	90.99 (2)
F6—Zr—F3^{i}	72.48 (3)	F2^{xiv}—K2—F4^{xiv}	57.253 (19)
F3—Zr—F3^{i}	64.52 (3)	F1^{v}—K2—F4^{xiv}	119.55 (2)
F5—Zr—K2^{ii}	88.23 (3)	F5^{xv}—K2—F4^{xiv}	80.80 (3)
F4—Zr—K2^{ii}	40.11 (2)	F2^{xiv}—K2—F6^{x}	88.34 (2)
F2—Zr—K2^{ii}	39.196 (16)	F1^{v}—K2—F6^{x}	89.34 (2)
F1—Zr—K2^{ii}	94.399 (18)	F5^{xv}—K2—F6^{x}	61.36 (2)
F6—Zr—K2^{ii}	118.17 (2)	F4^{xiv}—K2—F6^{x}	133.22 (2)
F3—Zr—K2^{ii}	106.885 (17)	F2^{xiv}—K2—F1	94.437 (19)
F3^{i}—Zr—K2^{ii}	168.149 (19)	F1^{v}—K2—F1	93.756 (18)
F5—Zr—K1^{iii}	145.96 (2)	F5^{xv}—K2—F1	169.73 (2)
F4—Zr—K1^{iii}	85.09 (2)	F4^{xiv}—K2—F1	88.93 (2)
F2—Zr—K1^{iii}	42.802 (17)	F6^{x}—K2—F1	127.72 (2)
F1—Zr—K1^{iii}	40.202 (17)	F2^{xiv}—K2—F7	119.06 (2)
F6—Zr—K1^{iii}	120.88 (2)	F1^{v}—K2—F7	67.88 (2)
F3—Zr—K1^{iii}	90.912 (19)	F5^{xv}—K2—F7	126.22 (3)
F3^{i}—Zr—K1^{iii}	107.430 (19)	F4^{xiv}—K2—F7	152.86 (3)
K2^{ii}—Zr—K1^{iii}	63.310 (5)	F6^{x}—K2—F7	69.22 (2)
F5—Zr—K1^{iv}	87.86 (2)	F1—K2—F7	64.05 (2)
F4—Zr—K1^{iv}	105.32 (2)	F2^{xiv}—K2—F7^{v}	117.85 (2)
F2—Zr—K1^{iv}	32.804 (16)	F1^{v}—K2—F7^{v}	63.44 (2)
F1—Zr—K1^{iv}	99.579 (17)	F5^{xv}—K2—F7^{v}	111.96 (2)
F6—Zr—K1^{iv}	174.36 (2)	F4^{xiv}—K2—F7^{v}	64.96 (2)
F3—Zr—K1^{iv}	42.430 (17)	F6^{x}—K2—F7^{v}	152.45 (2)
F3^{i}—Zr—K1^{iv}	104.196 (17)	F1—K2—F7^{v}	62.582 (19)
K2^{ii}—Zr—K1^{iv}	65.620 (5)	F7—K2—F7^{v}	101.78 (2)
K1^{iii}—Zr—K1^{iv}	64.248 (3)	F2^{xiv}—K2—F3^{xv}	116.502 (18)
F5—Zr—K2^{v}	146.34 (3)	F1^{v}—K2—F3^{xv}	55.698 (17)
F4—Zr—K2^{v}	123.85 (3)	F5^{xv}—K2—F3^{xv}	53.77 (2)
F2—Zr—K2^{v}	100.980 (17)	F4^{xiv}—K2—F3^{xv}	72.63 (2)
F1—Zr—K2^{v}	32.783 (18)	F6^{x}—K2—F3^{xv}	101.703 (19)
F6—Zr—K2^{v}	80.783 (19)	F1—K2—F3^{xv}	122.639 (18)
F3—Zr—K2^{v}	85.118 (18)	F7—K2—F3^{xv}	123.12 (2)
F3^{i}—Zr—K2^{v}	48.660 (19)	F7^{v}—K2—F3^{xv}	60.360 (19)
K2^{ii}—Zr—K2^{v}	125.009 (5)	F2^{xiv}—K2—K2^{v}	138.945 (17)
K1^{iii}—Zr—K2^{v}	63.039 (5)	F1^{v}—K2—K2^{v}	49.139 (14)
K1^{iv}—Zr—K2^{v}	100.533 (5)	F5^{xv}—K2—K2^{v}	139.29 (2)
F5—Zr—K2^{vi}	31.28 (2)	F4^{xiv}—K2—K2^{v}	109.46 (2)
F4—Zr—K2^{vi}	109.10 (3)	F6^{x}—K2—K2^{v}	117.109 (18)
F2—Zr—K2^{vi}	90.120 (17)	F1—K2—K2^{v}	44.617 (13)

F1—Zr—K2^{vi}	154.536 (18)	F7—K2—K2^{v}	53.362 (19)
F6—Zr—K2^{vi}	110.88 (2)	F7^{v}—K2—K2^{v}	48.418 (16)
F3—Zr—K2^{vi}	48.175 (19)	F3^{xv}—K2—K2^{v}	90.694 (14)
F3^{i}—Zr—K2^{vi}	84.499 (17)	F2^{xiv}—K2—Zr^{xiv}	29.024 (12)
K2^{ii}—Zr—K2^{vi}	95.611 (5)	F1^{v}—K2—Zr^{xiv}	148.434 (15)
K1^{iii}—Zr—K2^{vi}	128.158 (5)	F5^{xv}—K2—Zr^{xiv}	83.797 (19)
K1^{iv}—Zr—K2^{vi}	63.915 (5)	F4^{xiv}—K2—Zr^{xiv}	28.880 (14)
K2^{v}—Zr—K2^{vi}	127.033 (4)	F6^{x}—K2—Zr^{xiv}	114.644 (16)
F2^{vii}—K1—F4^{viii}	83.77 (2)	F1—K2—Zr^{xiv}	87.405 (13)
F2^{vii}—K1—F5^{ix}	93.40 (2)	F7—K2—Zr^{xiv}	138.215 (17)
F4^{viii}—K1—F5^{ix}	96.23 (2)	F7^{v}—K2—Zr^{xiv}	89.697 (17)
F2^{vii}—K1—F1^{x}	138.84 (2)	F3^{xv}—K2—Zr^{xiv}	97.590 (12)
F4^{viii}—K1—F1^{x}	136.82 (2)	K2^{v}—K2—Zr^{xiv}	124.431 (9)
F5^{ix}—K1—F1^{x}	89.00 (2)	F2^{xiv}—K2—K1^{xvi}	42.828 (13)
F2^{vii}—K1—F2^{x}	161.06 (2)	F1^{v}—K2—K1^{xvi}	129.466 (15)
F4^{viii}—K1—F2^{x}	81.60 (2)	F5^{xv}—K2—K1^{xvi}	40.607 (18)
F5^{ix}—K1—F2^{x}	76.358 (19)	F4^{xiv}—K2—K1^{xvi}	74.689 (18)
F1^{x}—K1—F2^{x}	58.032 (17)	F6^{x}—K2—K1^{xvi}	58.790 (16)
F2^{vii}—K1—F7^{xi}	72.91 (2)	F1—K2—K1^{xvi}	136.521 (14)
F4^{viii}—K1—F7^{xi}	145.75 (3)	F7—K2—K1^{xvi}	122.70 (2)
F5^{ix}—K1—F7^{xi}	109.61 (2)	F7^{v}—K2—K1^{xvi}	135.492 (17)
F1^{x}—K1—F7^{xi}	67.66 (2)	F3^{xv}—K2—K1^{xvi}	90.930 (13)
F2^{x}—K1—F7^{xi}	125.37 (2)	K2^{v}—K2—K1^{xvi}	175.841 (10)
F2^{vii}—K1—F3^{vii}	54.737 (17)	Zr^{xiv}—K2—K1^{xvi}	59.097 (4)
F4^{viii}—K1—F3^{vii}	77.52 (2)	Zr—F1—K2^{v}	122.73 (3)
F5^{ix}—K1—F3^{vii}	147.81 (2)	Zr—F1—K1^{iii}	111.05 (2)
F1^{x}—K1—F3^{vii}	117.33 (2)	K2^{v}—F1—K1^{iii}	103.28 (2)
F2^{x}—K1—F3^{vii}	132.321 (18)	Zr—F1—K2	128.62 (3)
F7^{xi}—K1—F3^{vii}	68.60 (2)	K2^{v}—F1—K2	86.244 (18)
F2^{vii}—K1—F7^{xii}	116.68 (2)	K1^{iii}—F1—K2	99.88 (2)
F4^{viii}—K1—F7^{xii}	66.87 (2)	Zr—F2—K1^{iv}	122.47 (3)
F5^{ix}—K1—F7^{xii}	142.08 (2)	Zr—F2—K2^{ii}	111.78 (2)
F1^{x}—K1—F7^{xii}	82.98 (2)	K1^{iv}—F2—K2^{ii}	110.29 (2)
F2^{x}—K1—F7^{xii}	67.95 (2)	Zr—F2—K1^{iii}	107.91 (2)
F7^{xi}—K1—F7^{xii}	101.41 (3)	K1^{iv}—F2—K1^{iii}	103.432 (19)
F3^{vii}—K1—F7^{xii}	64.47 (2)	K2^{ii}—F2—K1^{iii}	97.567 (19)
F2^{vii}—K1—F4^{ix}	71.922 (18)	Zr—F3—Zr^{i}	115.48 (3)
F4^{viii}—K1—F4^{ix}	135.74 (3)	Zr—F3—K1^{iv}	107.03 (2)
F5^{ix}—K1—F4^{ix}	50.694 (19)	Zr^{i}—F3—K1^{iv}	132.19 (3)
F1^{x}—K1—F4^{ix}	78.296 (19)	Zr—F3—K2^{vi}	100.81 (2)
F2^{x}—K1—F4^{ix}	111.259 (18)	Zr^{i}—F3—K2^{vi}	99.85 (2)

F7^{xi}—K1—F4^{ix}	59.63 (2)	K1^{iv}—F3—K2^{vi}	92.473 (18)
F3^{vii}—K1—F4^{ix}	113.649 (19)	Zr—F4—K1^{xvii}	138.12 (3)
F7^{xii}—K1—F4^{ix}	157.36 (2)	Zr—F4—K2^{ii}	111.01 (3)
F2^{vii}—K1—Zr^{x}	166.072 (14)	K1^{xvii}—F4—K2^{ii}	109.88 (2)
F4^{viii}—K1—Zr^{x}	109.69 (2)	Zr—F4—K1^{xviii}	99.42 (3)
F5^{ix}—K1—Zr^{x}	81.870 (17)	K1^{xvii}—F4—K1^{xviii}	89.73 (2)
F1^{x}—K1—Zr^{x}	28.747 (13)	K2^{ii}—F4—K1^{xviii}	89.27 (2)
F2^{x}—K1—Zr^{x}	29.286 (11)	Zr—F5—K2^{vi}	125.91 (4)
F7^{xi}—K1—Zr^{x}	96.241 (19)	Zr—F5—K1^{xviii}	125.86 (4)
F3^{vii}—K1—Zr^{x}	130.140 (14)	K2^{vi}—F5—K1^{xviii}	99.80 (2)
F7^{xii}—K1—Zr^{x}	73.412 (18)	Zr—F6—K2^{iii}	133.89 (3)
F4^{ix}—K1—Zr^{x}	95.194 (13)	K1^{xix}—F7—K2	95.21 (2)
F2^{vii}—K1—Zr^{vii}	24.729 (12)	K1^{xix}—F7—K1^{xii}	94.38 (3)
F4^{viii}—K1—Zr^{vii}	75.902 (18)	K2—F7—K1^{xii}	166.20 (3)
F5^{ix}—K1—Zr^{vii}	117.271 (17)	K1^{xix}—F7—K2^{v}	92.30 (2)
F1^{x}—K1—Zr^{vii}	137.870 (15)	K2—F7—K2^{v}	78.22 (2)
F2^{x}—K1—Zr^{vii}	154.647 (13)	K1^{xii}—F7—K2^{v}	91.52 (2)

Symmetry codes: (i) −*x*, −*y*, −*z*; (ii) −*x*+1/2, −*y*, *z*−1/2; (iii) *x*, −*y*+1/2, *z*−1/2; (iv) *x*, *y*−1, *z*−1; (v) −*x*, −*y*, −*z*+1; (vi) *x*, *y*, *z*−1; (vii) *x*, *y*+1, *z*+1; (viii) −*x*+1/2, −*y*+1, *z*+1/2; (ix) −*x*+1/2, *y*+1/2, *z*+1; (x) *x*, −*y*+1/2, *z*+1/2; (xi) −*x*, *y*+1/2, −*z*+3/2; (xii) −*x*, −*y*+1, −*z*+1; (xiii) −*x*+1/2, *y*+1/2, *z*; (xiv) −*x*+1/2, −*y*, *z*+1/2; (xv) *x*, *y*, *z*+1; (xvi) −*x*+1/2, *y*−1/2, *z*; (xvii) −*x*+1/2, −*y*+1, *z*−1/2; (xviii) −*x*+1/2, *y*−1/2, *z*−1; (xix) −*x*, *y*−1/2, −*z*+3/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
F7—H1···F6	0.87 (2)	1.52 (2)	2.3865 (10)	172 (2)

Fig. 1

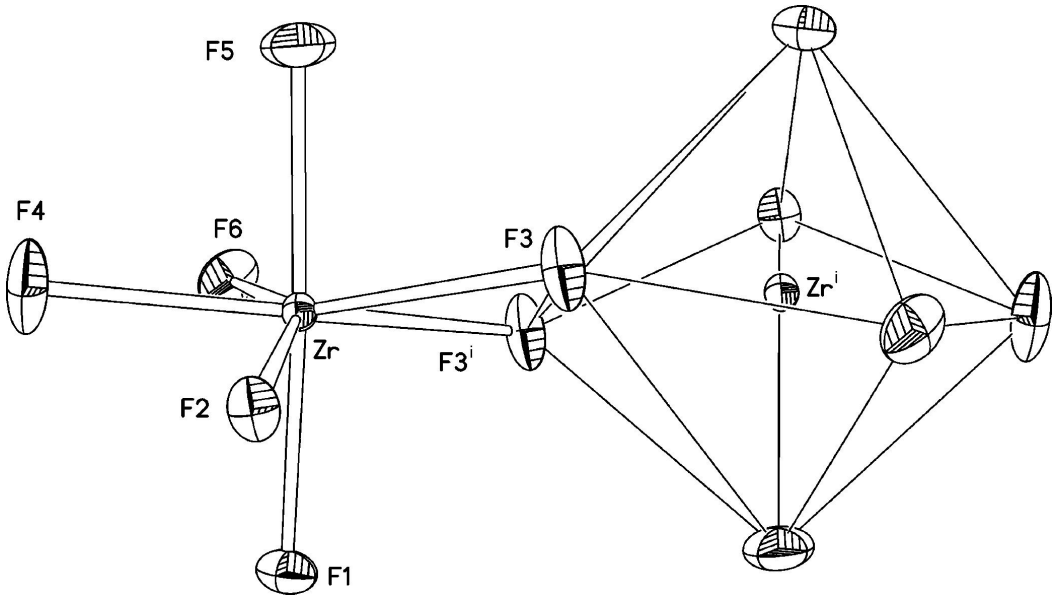

Fig. 2